



Solute diffusion of Ni in icosahedral Al–Pd–Mn quasicrystals

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ABSTRACT

This paper presents the results of an experimental study of Ni solute diffusion in the single-grain icosahedral quasicrystalline compound $\text{Al}_{70}\text{Pd}_{21}\text{Mn}_9$. Ni solute diffusion coefficients were measured over a temperature range from 699 to 1074 K. The radiotracer technique in combination with serial sectioning of the diffusion samples was used to determine concentration depth profiles. No deviation from Arrhenius behaviour was detected in the temperature range covered by the present experiments. The results are compared with the available diffusion data on quasicrystals and on diffusion in Al and various diffusion mechanisms proposed for quasicrystals are discussed. The most striking fact is that the diffusion of Ni and the diffusion of other transition-metal elements are found to be much slower than diffusion of non-transition-metal elements such as Ge and Zn in *i*-(Al–Pd–Mn) quasicrystalline alloys. Moreover, the activation enthalpies of diffusion of the transition-metal elements turned out to be considerably higher than those measured for the non-transition-metal elements. The present study shows that the diffusion of Ni atoms is likely to proceed by a vacancy mechanism via Al and Pd sites.

§1. INTRODUCTION

Quasicrystals are well ordered structures with a special crystallography because they possess a long-range translational order despite a non-crystallographic point group of symmetries. While the practical application of the bulk materials remains elusive until today, recent work has shown that coating of quasicrystals has intriguing properties. The properties which are especially promising include low coefficients of friction, good wear resistance and good corrosion resistance. Furthermore, quasicrystalline film can be prepared in an environmentally benign manner, which makes them an attractive alternative to conventional, chemically based platings for machine parts. Knowledge of the elementary process of atomic diffusion in quasicrystals is an important key for understanding the kinetics of many structural changes. However, diffusion data for quasicrystalline structures are still

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scarce. The few published experimental studies of diffusion in quasicrystals have been carried out on icosahedral Al–Pd–Mn (i-(Al–Pd–Mn)) and icosahedral Al–Cu–Fe quasicrystals. Kalugin and Katz (1993) predicted that, unlike bulk diffusion in metallic periodic crystals vacancies are not necessary for atomic motion in quasicrystalline structures. Owing to additional degrees of freedom of quasiperiodic systems one can construct an additional process for bulk diffusion in quasicrystals. Those defects are called dynamical phasons. The activation energy for such atomic jumps or phason flip is believed to be lower than that for the vacancy diffusion. In addition, a deviation of the diffusion coefficient from the normal Arrhenius law was predicted. Unfortunately, Kalugin and Katz were not able to determine the temperature at which these nonlinear Arrhenius behaviours are to be expected nor to estimate their order of magnitude. In this report we present the result of the diffusion measurements of ^{63}Ni in single-domain i-(Al–Pd–Mn) quasicrystals. The general diffusion behaviour will be discussed. Consideration will be given to published experimental results on diffusion and of the atomic structure of i-(Al–Pd–Mn) quasicrystals as obtained from neutron and X-ray diffraction studies.

§2. EXPERIMENTAL PROCEDURE

Cylindrical rods of i-(Al–Pd–Mn) quasicrystals were grown by a modified Bridgman technique in an Ar atmosphere using a crucible machined from Al_2O_3 at the Ames Laboratory, Iowa State University. Some details about the growth technique and the characterization have been given by Kycia *et al.* (1993). Cylindrical diffusion samples with single grains about 4–5 mm in diameter and 2 mm in thickness were cut from the rod with a Cu–diamond wire saw. The composition was checked by electron probe microanalysis for homogeneity and is $\text{Al}_{70}\text{Pd}_{21}\text{Mn}_9$. The single icosahedral structure was determined by Laue diffraction. After cleaning and polishing, all samples were pre-annealed in an Ar atmosphere at a temperature above the intended diffusion annealing temperature in order to eliminate possible surface strains and to reduce the dislocation density of the materials. In order to avoid evaporation losses from the sample surface, the polished sample was attached to a second sample dummy of i-(Al–Pd–Mn). Then both samples were wrapped in Ta foil and encapsulated in quartz ampoules in an Ar atmosphere and pre-annealed for 24 h. The quality of the surface of the samples was confirmed by conventional optical microscopy. The radioactive isotope used for the diffusion studies was ^{63}Ni . ^{63}Ni with a half-life of 100 years was purchased from E.I. DuPont de Nemours & Company as chloride solution. For diffusion experiments 6–10 kBq of the ^{63}Ni solution diluted with distilled water was dripped on to the polished sample surface and dried with an infrared lamp. The diffusion anneals were again performed under a purified Ar atmosphere in quartz tubes. The temperature was controlled by Pt–(Pt–Rh) thermocouples placed in close vicinity to the samples. The temperature uncertainty was evaluated to be about 2 K. For annealing times less than 4 h a heating-up correction was performed. After the diffusion anneal the sample was reduced in diameter with a specially designed grinding device by about $5(Dt)^{1/2}$ to eliminate lateral diffusion effects. The diffusion penetration profiles of the compound samples were determined by a serial sectioning technique using a precision parallel grinder with a special abrasive mylar foil (3M Company). The section thickness was calculated from the difference between the sample masses before and after grinding and from the diameter and density of the specimen. The sample weight was determined on a microbalance and the density by the Archimedes

method (5.05 g cm^{-3}). The low energy β -decays of the ^{63}Ni tracer were detected in a liquid scintillation counter, which provided a counting efficiency of about 90%. To achieve optimum counting conditions, the foil containing the radiotracer was dissolved in a counting vial in a sensitive scintillation cocktail. In this way any loss of activity was avoided and equal counting conditions for all sections were provided. The tracer concentration is proportional to the specific counting rate, which is the ratio of the counting rate to the section mass.

§ 3. RESULTS

Provided that the layer thickness of our deposits is less than $(Dt)^{1/2}/10$ the appropriate solution of the diffusion equation is well approximated by the thin film solution for a semi-infinite sample:

$$c(x, t) = \frac{M}{(\pi Dt)^{1/2}} \exp\left(-\frac{x^2}{4Dt}\right). \quad (1)$$

Here $c(x, t)$ is the averaged isotope concentration in a section parallel to the surface, M the initial tracer concentration on the surface, x the penetration depth and t the annealing time. The calculated diffusion coefficient D represents the true volume diffusion. For sections which are thin compared with the thickness of the tracer layer a plot of $\log c$ versus x^2 should be linear with slope $-1/(4Dt)$. Most of the experiments indeed show Gaussian penetration profiles with a decrease in tracer concentration for about two to three orders of magnitude. This indicates that the boundary conditions inherent to equation (1) are well fulfilled. The concentration profiles at high temperatures are shown in figure 1. The solid lines are fits to equation (1). At low temperatures penetration profiles reveal deviations from Gaussian behaviour. Instead of equation (1), the complementary error function

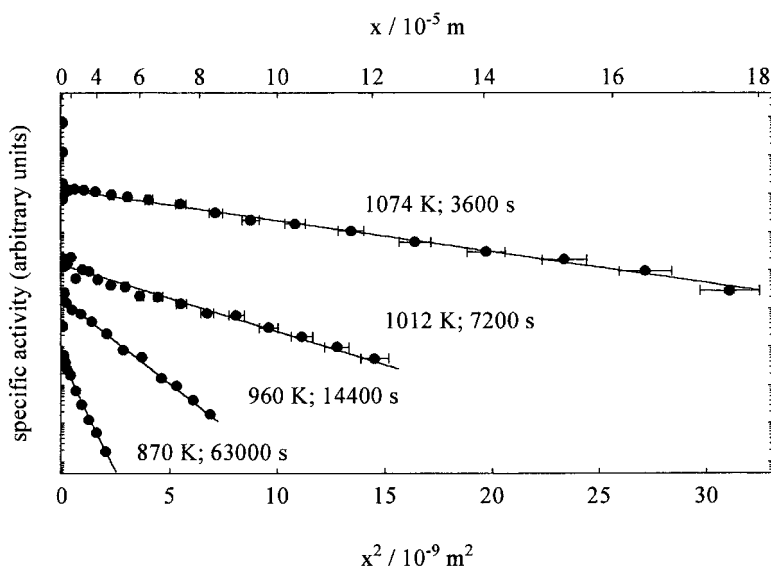


Figure 1. Penetration profiles for diffusion of ^{63}Ni in *i*-(Al-Pd-Mn) at temperatures higher than 869 K: (—), fits by equation (1).

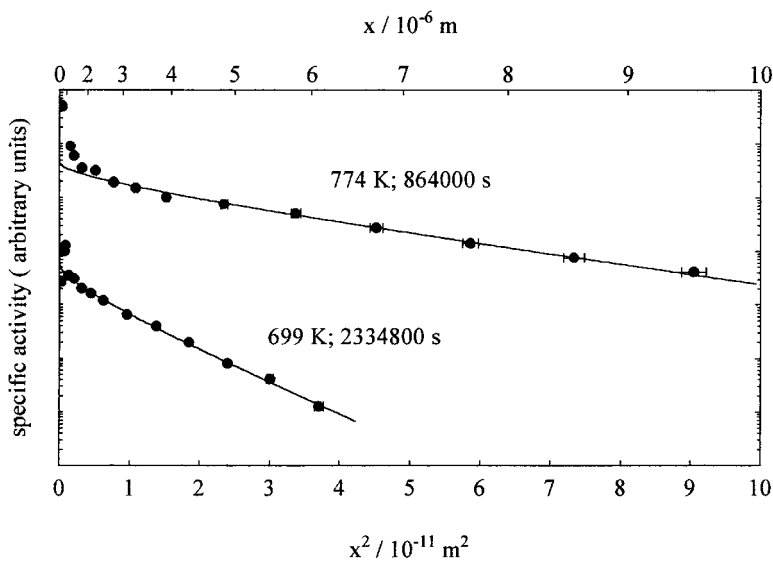


Figure 2. Penetration profiles for diffusion of ^{63}Ni in i-(Al-Pd-Mn) at temperatures lower than 775 K : (—), fits by equation (2).

was applied to the data points:

$$c(x,t) = C_0 \operatorname{erfc}\left(\frac{x}{2(Dt)^{1/2}}\right). \tag{2}$$

In equation (2), C_0 denotes the solubility limit and erfc the complementary error function. The solid curves in figure 2 represent the fit according to equation (2). The deviations might result from a Ni rich intermetallic compound formed in the near-surface region or from the formation of an oxide layer acting as a diffusion barrier. These phenomena have already been observed in earlier diffusion experiments on Al and on Al -base alloys (Beke *et al.* 1976). The diffusion coefficients deduced from the fits are listed in table 1. D_1 and D_2 in table 1 represent the diffusion coefficients obtained by fitting equation (1) and equation (2) to the diffusion profiles.

Table 1. Diffusion coefficients of ^{63}Ni in i-(Al-Pd-Mn) .
 D_1 and D_2 are the values obtained by fitting equation (1) and equation (2) respectively to the diffusion profiles.

T (K)	t (s)	D_1 ($\text{m}^2 \text{ s}^{-1}$)	D_2 ($\text{m}^2 \text{ s}^{-1}$)
699	2 334 800	6.96×10^{-19}	8.31×10^{-19a}
774	864 000	3.04×10^{-17}	4.06×10^{-17a}
870	63 000	1.68×10^{-15a}	
960	14 400	2.34×10^{-14a}	
1012	7 200	7.96×10^{-14a}	
1074	3 600	3.63×10^{-13a}	

^a D values used to the least-squares fits of the Arrhenius relation.

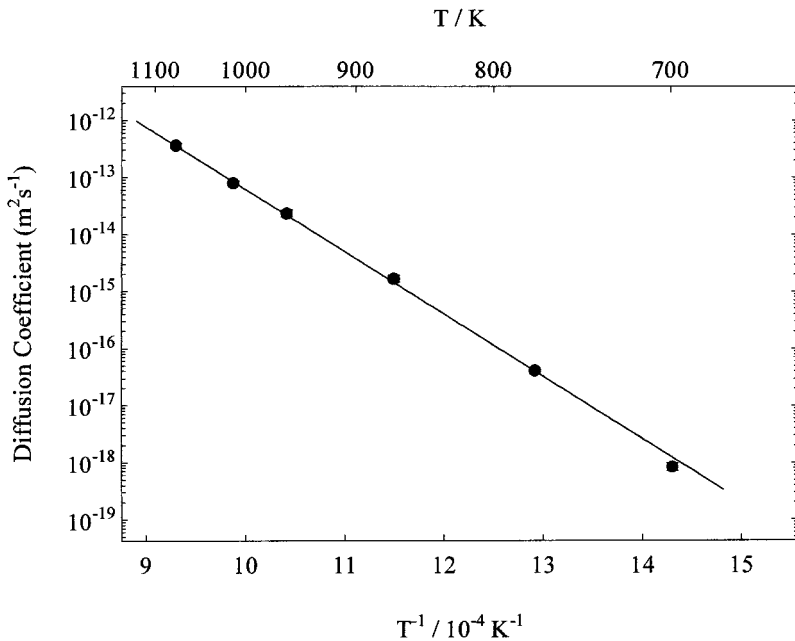


Figure 3. Arrhenius diagram of ^{63}Ni solute diffusion in i-(Al-Pd-Mn) single quasicrystals.

Least-squares fits of the Arrhenius relations to D values indicated with a superscript a in table 1 yield the following expression for Ni diffusion in the range from 699 to 1074 K:

$$D = 5.3 \times 10^{-3} \exp \left(- \frac{209.0 \pm 0.4 \text{ kJ mol}^{-1}}{RT} \right) \text{ m}^2 \text{ s}^{-1}. \quad (3)$$

In this equation, R denotes the gas constant and T the absolute temperature. The resulting diffusion coefficients of ^{63}Ni in $\text{Al}_{70}\text{Pd}_{21}\text{Mn}_9$ single quasicrystals are plotted in an Arrhenius diagram presented in figure 3.

§ 4. DISCUSSION

The Ni diffusion was studied in the present system to obtain a more comprehensive picture of diffusion in i-(Al-Pd-Mn) quasicrystals. Figure 4 presents an Arrhenius plot of the volume diffusion coefficients available for single-domain i-(Al-Pd-Mn). Data points of the present study together with the fitted Arrhenius line are shown in the same figure. In table 2 the Arrhenius parameters of all currently available diffusion experiments in single i-(Al-Pd-Mn) quasicrystals are summarized.

An inspection of the table shows that for most of the elements no indication of a deviation from the Arrhenius behaviour was found above 700 K or for diffusion coefficients higher than $1 \times 10^{-18} \text{ m}^2 \text{ s}^{-1}$. Also it appears that the diffuser elements can be classified into two groups: slow and fast diffusers. Both experimental findings are discussed in some detail in the following.

The first measurements of Mn and Fe diffusion on single quasicrystals were performed by Zumkley *et al.* (1996) and simultaneously Mn diffusion by

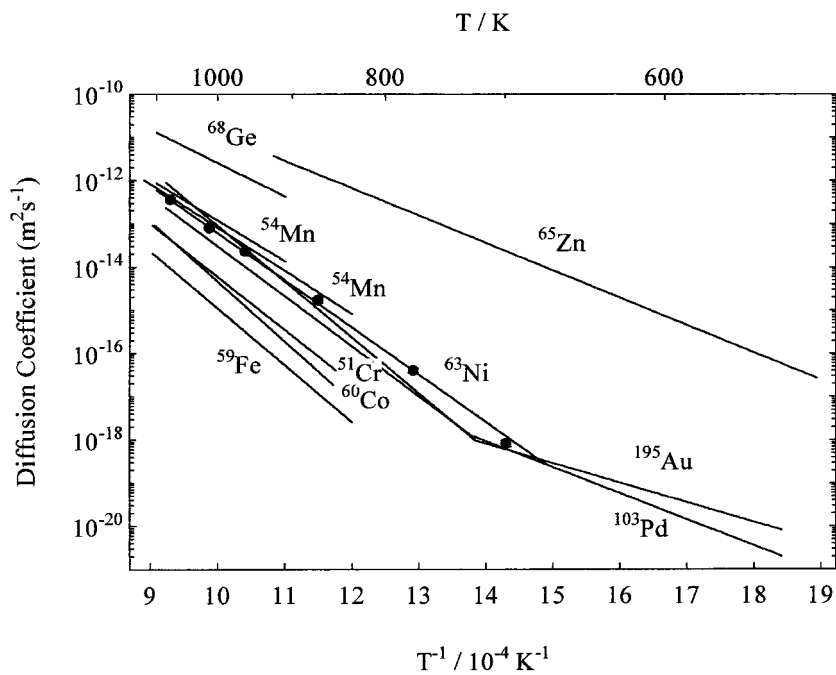


Figure 4. Temperature dependence of the diffusion coefficients of various metal elements in i-(Al-Pd-Mn) single quasicrystals. For references see text.

Table 2. Arrhenius parameter of various metal elements in i-(Al-Pd-Mn) single quasicrystals.

Isotope	Q (kJ mol ⁻¹)	D_0 (m ² s ⁻¹)	T (K)	Reference
Fast diffusion via vacancies				
⁶⁸ Ge	148.0	1.4×10^{-4}	908–1100	Sprengel <i>et al.</i> (1997)
⁶⁵ Zn	121.3	2.9×10^{-5}	528–923	Mehrer <i>et al.</i> (1998)
Slow diffusion via vacancies				
⁶³ Ni	209.0	5.3×10^{-3}	699–1074	Present work
¹⁹⁵ Au	248.0	8.2×10^{-1}	723–1083	Blüher <i>et al.</i> (1998)
¹⁰³ Pd	223.8	1.2×10^{-2}	723–1083	Blüher <i>et al.</i> (1998)
⁶⁰ Co	266.5	3.9×10^{-1}	853–1103	Sprengel <i>et al.</i> (1998a)
⁵⁹ Fe	251.8	1.6×10^{-2}	833–1132	Zumkley <i>et al.</i> (1996)
⁵⁴ Mn	180.8	3.4×10^{-4}	908–1100	Sprengel <i>et al.</i> (1996)
	192.0	8.9×10^{-4}	833–1123	Zumkley <i>et al.</i> (1996)
⁵¹ Cr	235.5	1.2×10^{-2}	850–1107	Sprengel <i>et al.</i> (1998b)
Phason-assisted diffusion				
¹⁹⁵ Au	86.4	4.6×10^{-1}	543–723	Blüher <i>et al.</i> (1998)
¹⁰³ Pd	115.8	1.4×10^{-10}	543–723	Blüher <i>et al.</i> (1998)
Phason diffusion in a restricted space				
²⁷ Al	1 meV		12–72	Dolinsek <i>et al.</i> (1999)

Sprenkel *et al.* (1996) in the same system. In the meantime, the diffusion of Ge (Sprenkel *et al.* 1997), Co (Sprenkel *et al.* 1998a) and Cr (Sprenkel *et al.* 1998b) was also studied, while Mehrer *et al.* (1998) conducted a further study on Zn diffusion. In all these experiments the diffusion data could be described by only one set of Arrhenius parameter. Diffusion data published by Blüher *et al.* (1998) on the diffusion of Pd and Au in i-(Al-Pd-Mn) single quasicrystals between 543 and 1083 K showed at lower temperatures (below 723 K) a strong deviation of the low-temperature diffusion coefficients from those extrapolated from higher temperatures. For the low-temperature region, distinctly lower activation energies and pre-exponential factors were calculated.

Recently Dolinsek *et al.* (1999) studied the Al self-diffusion in i-(Al-Pd-Mn) at temperatures between 4 and 400 K by ^{27}Al nuclear magnetic resonance (NMR) measurements. In the temperature range between 12 and 72 K they obtained an unusually small activation energy of 1 meV. In periodic crystals such as pure metals and intermetallic compounds the bulk diffusion mainly results from the presence of lattice vacancies. The diffusion is thermally activated with a total energy that includes the energy of vacancy creation and the activation energy of an atomic jump. For these systems the total activation energy is usually found to be of the order of 100–300 kJ mol $^{-1}$ (Philibert 1991). The activation enthalpies Q and pre-exponential factors D_0 for diffusion in i-(Al-Pd-Mn) quasicrystals at high temperatures are essentially in the same range.

Positron lifetime spectroscopy experiments on i-(Al-Pd-Mn) (Kanazawa *et al.* 1997) confirmed the existence of structural vacancies probably on Al sites. Furthermore, Beeli *et al.* (1998) observed that the faceted microholes in i-(Al-Pd-Mn) formed by condensation of thermal vacancies grow via classical Ostwald ripening at high temperatures between 1023 and 1103 K. Thus, the holes are believed to grow by a classical diffusion-controlled ripening process.

Measurements of the activation volume of Mn (Zumkley *et al.* 1997) and of Zn (Mehrer *et al.* 1998) obtained from the pressure dependence of diffusion seemed to indicate that bulk diffusion in icosahedral (Al-Pd-Mn) quasicrystals at least in the high temperature range occurs by a vacancy mechanism. The fixed activation volumes are similar to those which are found for self-diffusion in fcc metals.

The diffusivities at the melting point of i-(Al-Pd-Mn) ($T_M = 1159$ K (Beeli and Nissen 1993)) and the activation energies are plotted in figure 5 versus the position of the elements in the periodic table (valence).

A close inspection of table 2 and figure 5 suggests that the diffusion data can be separated into two groups (for example Mehrer *et al.* (1998)). The so-called fast diffusers in i-(Al-Pd-Mn) together with Zn and Ge have relatively small pre-exponential factors and low activation energies. The activation energies of the latter are 1.28 and 1.53 eV respectively. The elements of the second group diffuse with much lower rates in i-(Al-Pd-Mn) and their activation energies are much larger.

The following discussion is an attempt to explain the different diffusion behaviours of both groups on the basis of the cluster model for i-(Al-Pd-Mn) quasicrystals (Janot 1996). The structure as obtained from diffraction data (Boudard *et al.* 1992) has been described in terms of stable clusters and a self-similar arrangement (Janot and de Boissieu 1994, Janot 1996). Essentially the structure is based on atomic units of 51 atoms, called pseudo-Mackay icosahedra (PMIs). Each cluster consists of two centrosymmetrical shells, an inner small centred core of nine atoms, an icosahedron (12 atoms) and an icosidodecahedron (30 atoms). The last two shells have

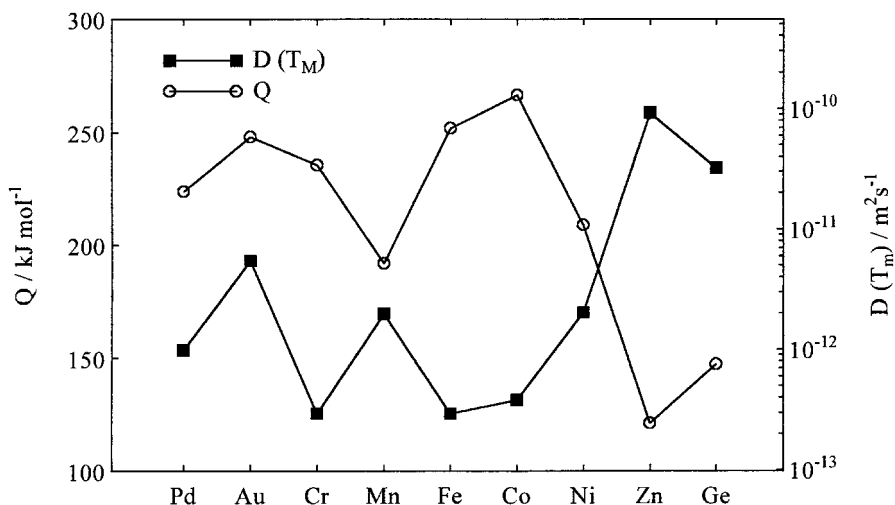


Figure 5. Diffusivities at the melting point of *i*-(Al-Pd-Mn) ($T_M = 1132$ K) and activation enthalpies Q of various metal elements in *i*-(Al-Pd-Mn).

nearly the same radii and form altogether the external shell. The external shell is τ (inflation scale factor) times larger than the inner shell. Three cluster, namely PMI-A, PMI-T(Pd) and PMI-T(Mn) with identical geometries but different chemical compositions have been identified so far in the structure. The PMI-A contains 38 Al, seven Pd and six Mn atoms, the PMI-T(Pd) 31 Al plus 20 Pd atoms, and the PMI-T(Mn) 30 Al plus 21 Pd atoms. All sites of the larger shell are occupied but the small inner shell is a piece of a pentagonal dodecahedron whose 20 atomic sites are only partially occupied. This incompletely filled inner shell is the reason that the three different clusters are able to overlap.

We propose that the relatively fast diffusion of Zn and Ge in the high temperature region occurs via vacant sites on the Al network of the clusters. Two arguments leading to this proposal may be mentioned. In the first place, there exists a high number of structural vacancies on Al sites, which will largely favour diffusion via the Al network. In addition, the elements Zn and Ge are relatively fast diffusers in both pure Al and *i*-(Al-Pd-Mn) quasicrystals. The quasicrystalline compound consists of more than 70 at.% Al. The values calculated for the pre-exponential factors and the activation energies are very similar for various materials and also lie in the range of the activation energy determined for Al self-diffusion in pure Al of 123.5 kJ mol⁻¹ (Dais *et al.* 1987). In contrast with Zn and Ge diffusion the transition metal elements Co, Fe, Mn, Cr, Au and Pd are slow diffusers in *i*-(Al-Pd-Mn) which have activation energies ranging from 192 to 266 kJ mol⁻¹ and higher pre-exponential factors. The Ni diffusivity is of the same magnitude as the diffusivities of Mn, Au or Pd in *i*-(Al-Pd-Mn). Similar to diffusion in pure Al the respective 3d transition metal elements are diffusers with high activation enthalpies and pre-exponential factors in *i*-(Al-Pd-Mn). The heavier metals Au and Pd diffuse with similar rates in *i*-(Al-Pd-Mn) and are to be added to the group of slow diffusers. Unfortunately only the Au diffusion was determined in pure Al. Its diffusivity is several orders of magnitude higher than in the icosahedral structure. Since both elements are chemically similar and their diffusivities in *i*-(Al-Pd-Mn) are about the same, we suppose that the Pd diffusion

will occur with similar rates to Au diffusivity in Al. Nevertheless it seems that the diffusion of slow diffusing elements is hindered inside of the quasicrystalline structure. It has been found that the nearest neighbour distances in quasicrystals are 5% shorter than in the corresponding metals. This will especially affect the diffusion behaviour of larger atoms as Pd or Au. Blüher *et al.* (1998) predicted the following mechanism for slowly diffusing elements in i-(Al-Pd-Mn). They supposed these elements to migrate via vacant sites of the Al and Pd substructure of the icosahedral structure in which the diffusivity is slow in the Pd region and thus Pd site controlled.

Besides normal vacancies a second type of dynamical defect exists in quasicrystals, called phasons. Phasons arise as a result of violations of the matching rules. Their study is important for understanding many basic issues in quasicrystalline compounds related to the stability of the structure in the random tiling model or phase transitions. Kalugin and Katz (1993) suggested a possible mechanism for bulk self-diffusion in quasicrystals based only on the existence of dynamical phasons. They expect a strongly nonlinear Arrhenius behaviour for diffusion in quasicrystals. At low temperatures, phason assisted diffusion should be the predominant mechanism because the distances of phason related atomic jumps in quasicrystals are shorter than for conventional atom jumps over vacancies. For this reason the activation energy of an atomic jump or phason flip is believed to be lower than in metals with periodic structure. Computer simulations based only on phason defects in a two- and three-dimensional quasiperiodic arrangement confirmed that in an aperiodic structure the phason mechanism can result in long-range diffusion (Joseph *et al.* 1994, Ebinger *et al.* 1998). Unfortunately, no other possible mechanism besides the phason mechanism was included in their models. A molecular dynamics study (Roth and Gähler 1997) indicated that the phason mechanism needs to be catalysed by half-vacancies in order to be effective. Also they confirmed that the contributions of phason diffusion and vacancy diffusion were difficult to separate. Because of the great number of rather complex parameters none of the theoretical models has so far been able to give some quantitative details about diffusivities and activation energies.

Two recent atomic motion studies were performed both on i-(Al-Pd-Mn) (Dolinsek *et al.* 1999) and on i-(Al-Re-Pd) (Dolinsek *et al.* 1998) quasicrystals at relatively low temperatures below 400 K by ^{27}Al NMR. These workers claimed that they had found the first proof for the existence of dynamical phasons in such a low temperature regime. In case of the i-(Al-Pd-Mn) alloy they represented their measurements by different Arrhenius laws above and below 72 K. For the low region they obtained a very small activation enthalpy of 96 J mol^{-1} which is of an order of magnitude comparable with diffusion in liquids. Dolinsek *et al.* assumed diffusion in this temperature range to take place solely in a restricted space where the number of phason defects is small. At the percolation threshold of 72 K the phason density is large enough and long-range diffusion can start via phason flips and increases strongly. The fact that Dolinsek *et al.* (1999) attributed some local arrangement of atoms in a spatially limited range to self-diffusion can be criticized. Self-diffusion in its classical sense implies long-range transport of atoms. Furthermore these workers have failed to produce clear evidence that the experimentally observed atom movements are caused by phason flips. Also Blüher *et al.* (1998) found for temperatures below 723 K for Au and Pd diffusion in i-(Al-Pd-Mn) a deviation from the Arrhenius equation with lower activation energies and pre-exponential factors than in the high temperature region, but much higher than in the low temperature

range of Dolinsek *et al.* Blüher *et al.* suggested that in this temperature range the diffusion of Au and Pd atoms occurs by vacancies which can be triggered by phasons. In contrast to this result, in the present study we measured Ni diffusion down to 699 K and found no deviation at these low temperatures. The diffusivity of Ni was found to be comparable with the diffusivity of Au and Pd in i-(Al-Pd-Mn) at high temperatures. A further diffusion study of Zn performed from 923 to 523 K by Mehrer *et al.* (1998) also showed no such deviation and no indication of phason-related diffusion.

From our results it seems difficult to identify any phason contributions to diffusion. Therefore we suggest on the basis of the above-mentioned reasons that the diffusion of Ni atoms occurs only via vacancies in the entire temperature range.

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